

**S2 Fig.** (A) MALDI-TOF MS spectra of substrate **11** (sequence Asp-Ala-Pro-Asp). (B) After incubation (20  $\mu$ M) with Caspase-3 for 2 h. (C) After incubation (20  $\mu$ M) with Caspase-7 for 2 h (detected as the PBS adduct.)

Caspase-3 or caspase-7 (R&D systems, USA) was added to 100  $\mu$ L of caspase assay buffer (final enzyme concentration 0.4  $\mu$ M) with the substrates at 10–20  $\mu$ M. The samples were incubated in Eppendorf Thermomixer® Comfort shaking at 1200 rpm for 2h at 37 °C. MALDI-TOF mass spectra were recorded as described in Table S1 .